TITLE: BIOCHEMICAL OXYGEN DEMAND

MATRICES:

This SOP pertains to aqueous matrices.

DETECTION LIMITS:

Reporting Limits are used in the absence of Project or State Specific Required Detection Limits.

The method detection limits (MDL) determination is not applicable to this method.

See Appendix B Table A for a summary

1.0 **SCOPE AND APPLICATION:**

1.1 The biochemical oxygen demand test is used for determining the relative oxygen requirements of municipal and industrial wastewaters. Applying this test to the discharge of organic wastes, it is possible to calculate the effect of the discharge on the oxygen resources of the receiving water. Data from BOD tests are used for developing engineering criteria when designing wastewater treatment plants.

1.2 The BOD test is an empirical procedure which measures the oxygen required for the biochemical degradation of organic matter and the oxygen used to oxidize inorganic material such as sulfides and ferrous iron. (carbonaceous demand)

1.3 This test also may measure the oxygen used to oxidize reduced forms of nitrogen. (nitrogenous demand)

2.0 **SUMMARY OF THE TEST METHOD:**

2.1 The sample of waste, or an appropriate dilution, is incubated for 5 days at 20^0 C, in the dark. The reduction in the dissolved oxygen concentration during the incubation period yields a measure of the biochemical oxygen demand

3.0 **DEFINITIONS**:

3.1 Definitions of terms used throughout this document are defined in Appendix A.

4.0 **INTERFERENCES:**

4.1 pH values outside the range of 6.5 to 7.5 To correct for this, use sulfuric acid or sodium hydroxide to bring the pH into the proper range.

4.2 Residual chlorine can be destroyed by adding sodium sulfite (Na2SO3) to the sample.

4.3 The temperature of the samples should be 20^0C+/- 3^o C when performing the analysis.

4.4 Samples containing heavy metals often require special study and treatment.

5.0 **SAFETY:**

5.1 Safety is everyone's business at En Chem, Inc. and everyone is responsible for assisting in reducing unsafe and unhealthy working conditions or potential hazards. The company provides you with a safe place to work, but we need your cooperation to keep it safe. When you see something that does not look safe, or you see someone working in an unsafe manner, mention it to your Supervisor.

5.2 All samples should be treated as hazardous. Safety glasses, gloves, and lab coats are to be worn. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by a safe technique. A reference file of material safety data sheets should also be made available to all personnel involved in the chemical analysis.

5.3 Required Safety Equipment is listed in Appendix B Table B.

6.0 **EQUIPMENT AND SUPPLIES:**

6.1 pH meter and buffers. (see pH SOP for instructions)

6.2 Dissolved oxygen meter: YSI Model 5000 with YSI 5010 stirring probe.

6.3 Adjustable pipetter, various sized wide bore pipettes, pipette bulb.

6.4 50, 100, and 250 mL graduated cylinders.

6.5 Incubators set at 20^0C +/- 1^0C (Fischer Low Temperature Incubator Model 307, VWR Scientific Low Temperature Incubator Model 2020, and Precision Low Temperature Incubator.)

6.6 300 mL glass BOD bottles with ground glass or plastic stoppers and water seal cap.

6.7 Glass microfiber filters (Fisher G6 Cat.# 09-804-90A), for use in dissolved BOD's.

6.8 Water bath set at 20^0C.

6.9 See Appendix B Table C and Table D for a summary.

7.0 **REAGENTS AND STANDARDS:**

7.1 Phosphate Buffer Solution:

Dissolve 8.5 g potassium phosphate monobasic (KH2PO4), 21.75 g potassium phosphate dibasic (K2HPO4), 33.4 g sodium phosphate dibasic (Na2HPO4 .7H2O), and 1.7 g ammonium chloride (NH4Cl) in 500 mL distilled water and dilute to 1000 mL. Store in refrigerator. The pH should be 7.2 without further adjustment. Discard if biological growth is present.

Alternatively, use a commercially prepared phosphate buffer - Fisher#: SP341-1.

7.2 Magnesium Sulfate:

Dissolve 22.5 g magnesium sulfate (MgSO4 .7H2O) in distilled water and dilute to 1000 mL.

Alternatively, use commercially prepared magnesium sulfate - Fisher#: SM109-1.

7.3 Calcium Chloride:

Dissolve 27.5 g anhydrous calcium chloride (CaCl2) in distilled water and dilute to 1000 mL.

Alternatively, use commercially prepared calcium chloride - VWR#: VW3308-1.

7.4 Ferric Chloride:

Dissolve 0.25 g ferric chloride (FeCl3 .6H2O) in distilled water and dilute to 1000 mL.

Alternatively, use commercially prepared ferric chloride - VWR#: VW3318-1.

7.5 Sodium Sulfite, anhydrous (Na2SO3)

7.6 Glucose/Glutamic Acid Standard (198 +/- 30.5 mg/l):

Dissolve 150 mg glutamic acid and 150 mg glucose in distilled water and dilute to 1000 mL. (must be made fresh daily)

7.7 Nitrification Inhibitor:

Nitrification inhibitor HACH Formula 2533 (TCMP).

7.8 Starch Indicator Paper

7.9 Sodium Hydroxide, 1N:

Dissolve 4 g of sodium hydroxide, NaOH, in 80 mL distilled water. When cool dilute to 100 mL.

7.10 Sulfuric Acid 1 N:

Dilute 2.8 mL concentrated sulfuric acid, H2SO4, to 100 mL with distilled water.

CAUTION: Always add acid to water, NEVER water to acid. This reaction liberates excessive heat and may react violently.

7.11 Distilled Water

Store distilled water as necessary for quality improvement. Store in BOD incubator for at least 12 hours prior to use.

7.12 Dilution Water

Add 1.0 mL magnesium chloride (7.2), 1.0 mL calcium chloride (7.3), 1.0 mL ferric chloride (7.4) and 1.0 mL phosphate buffer (7.1) for each 1000 mL of distilled water.

Note: The dissolved oxygen level of this water should be between 8.3 - 8.9 mg/L prior to use. If the D.O. exceeds this range proceed to section 8.1.

7.13 See Appendix B Table E and Table F for a summary.

8.0 **SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE:**

8.1 Sample Collection is done with clean glass or plastic bottles.

8.2 Sample Preservation consists of cooling samples at 4.0^0 to 6.0^0 C in shipment and storage prior to analysis .

8.3 Samples must be analyzed within 48 hours from the end of the sampling period.

8.4 See Appendix B Table G for a summary.

9.0 **QUALITY CONTROL:**

9.1 Prepare two bottles with dilution water only. These blanks will be used to determine the amount of dissolved oxygen in the dilution water, the quality of the dilution water and the cleanliness of the BOD bottles.

9.2 The D.O. difference should be no more than 0.2 mg/L. If the D.O. difference is greater than 0.2 mg/L, recalibrate the meter. If the D.O. difference is still out of range, bleach all of the containers that hold distilled or diluted water. If the D.O. difference is still out of range, look for other sources of contamination.

Note: Bleaching these containers should be done upon emptying them and before refilling to prevent this from happening. The carboys are also allowed to air dry before refilling.

9.3 Glucose/Glutamic Acid Standards are set at the beginning of the batch and after every 10 samples. The BOD of the standards must be within 198+/-30.5 mg/L (167.5-228.5 mg/L).

9.4 Duplicate samples are set every 10 samples. The RPD between the results must be less than or equal to 20%.

9.5 See Appendix B Table H for a summary of frequency.

10.0 **CALIBRATION AND STANDARDIZATION**:

10.1 The dissolved oxygen probe membrane is replaced weekly on average. However, should bubbles form under the membrane, the membrane must be changed. The probe electrode is cleaned monthly by soaking it in a 15 % v/v ammonium hydroxide solution for 10 minutes.

10.2 The dissolved oxygen meter must be calibrated daily (2x/day). Check membrane to see if it needs to be replaced, dry off membrane, and let probe come to equilibrium for 1/2 hour in BOD bottle filled with one inch of distilled water. The meter is calibrated in water-saturated air following instructions in the operations manual.

10.3 Clean bottles in dishwasher with Lab Solutions Powder detergent and rinse thoroughly with de-ionized water. Bottles should be completely dry before use.

10.4 Dilution H2O is initially checked before setting up blanks: one carboy will fill about 1.25 aspirator bottles. These bottles can be filled to a volume that is needed for that day's set-up. One BOD bottle is filled from each aspirator bottle and the D.O. is determined. If the D.O. is not with 8.3 to 8.9 mg/L, proceed with one of the following:

o If the D.O. is < 8.3 mg/L, shake the aspirator bottle until the D.O is within 8.3 to 8.9 mg/L.

o If the D.O. is > 8.9 mg/L, pour 2-3 L of the water to a 4000 mL beaker and heat on a hot plate until D.O. is reduced. Pour the water back into the aspirator bottle. Pour a portion of the water from the aspirator bottle to a BOD bottle. Read the BOD bottle. If the D.O. is not within 8.3 to 8.9 mg/L, repeat this step. Use the water when the temperature has returned to 20°C. Alternately, the water can be filtered using Fisher G6 Cat.# 09-804-90A, glass microfiber filters.

o If one of the aspirator bottles has a high D.O. and one a low D.O., mix a portion (4-6 L) together and return the water to the aspirator bottle. Pour a portion of this water into a BOD bottle. Read the BOD bottle, if the D.O. is within 8.3 to 8.9 mg/L, proceed with the analysis.

Note: The temperature at which the distilled water is stored should be kept at 20 to 25^{o} C to minimize the above steps.

10.5 Prepare two bottles with dilution water only, these blanks will be used to determine the amount of dissolved oxygen in the dilution water, the quality of the dilution water and the cleanliness of the BOD bottles.

10.6 The D.O. difference should be no more than 0.2 mg/L. If the D.O. difference is greater than 0.2 mg/L, recalibrate the meter. If the D.O. difference is still out of range, bleach all of the containers that hold distilled or diluted water. If the D.O. difference is still out of range, look for other sources of contamination.

Note: Bleaching these containers should be done upon emptying them and before refilling to prevent this from happening. The carboys are also allowed to air dry before refilling.

10.7 Glucose/Glutamic Acid Standards are used to determine the quality of the dilution water and accuracy of the D.O. meter

10.8 Seed Correction is accomplished by setting up 4 bottles with 3 mL, 5 mL, 10 mL and 20 mL of undiluted seed and fill with dilution water. By determining the BOD of the seed, the seed correction factor can be calculated for each seeded sample.

NOTE: Polyseed may be substituted if a supply of treatment plant seed is unavailable.

11.0 **PROCEDURE:**

11.1 For Carbonaceous BOD add 0.16 mg of Nitrification inhibitor (7.7) to each bottle, prior to following the procedure for total BOD as outlined starting in 11.3.

11.2 For Dissolved BOD filter the sample through a glass fiber , prior to following the procedure for total BOD as outlined starting in 11.3.

For all BOD analysis, Glucose/Glutamic Acid Standards are set at the beginning of the batch and after every 10 samples with 6 mL of Glucose/Glutamic acid standard (7.6) to a BOD bottle The bottle is then filled to capacity. (~ 300 mL)

For all BOD analysis by matrice, duplicates are set 1 per 10 samples.

For Total BOD follow the steps for analysis as outline starting in 11.3

11.3 For Total BOD follow the procedure below.

11.4 Neutralize Sample pH

- Measure and record the pH of the sample in the pH analysis logbook. If the sample does not fall within the range of 6.5 to 7.5, neutralize the sample into this range with sodium hydroxide (reagent 7.9), or sulfuric acid (reagent 7.10), whichever is appropriate.
- If the pH is within these limits proceed to section 11.5.

11.5 Check for Residual Chlorine

- Immerse a piece of potassium iodine (KI) starch indicator paper into all samples.
- If the indicator paper shows a positive chlorine result, remove chlorine by adding enough sodium sulfite (Na2SO3) (6.5) while stirring, to eliminate all of the chlorine. Refer to the chlorine residual procedure for specifics on this procedure. Make sure that enough sample is dechlorinated to cover the volume needed for sample dilutions. Samples corrected for chlorine must be seeded. (see section 11.7)
- If the indicator paper shows a negative result, proceed with analysis as given below.
- Warm samples to 20^{o} C +/- 3^{o}C.
- Place samples in a water bath set at 20^{o} C for a sufficient amount of time to bring their temperature up to to 20^{o} C +/- 3^{o}C.

11.7 Seeding

- It is necessary to have present a population of microorganisms capable of oxidizing the biodegradable organic matter in a sample. The seed is settled domestic wastewater at 20^{0} C 1 to 36 hours old.
- Seed must be added to untreated industrial samples, high temperature samples, disinfected samples, samples with extreme pH values and any samples that were dechlorinated. All other samples need not be seeded; whenever unsure seed the sample.
- Add 2.0 mL of seed to each BOD bottle requiring seeding.

11.8 Sample Volumes

- Estimate the BOD of the sample and select dilutions that will yield a D.O. difference of at least 2.0 mg/L and a final D.O. of at least 1.0 mg/L, using the following as a guideline. (Set up at least three dilutions per sample)

Estimated BOD (mg/L)	Sample Volumes
Less than 10 (river water)	50, 100, 200
10 - 30 (treated effluent)	25, 50, 100
30 - 60	15, 25, 50
60 - 90	10, 15, 25
90 - 150	5, 10, 15
150 - 300 (raw wastewater)	3, 5, 10
300 - 700 (industrial waste)	1, 3, 5
700 - 1500	0.5, 1, 3
1500 - 2500	0.3, 0.5, 1

Note: If greater than 150 mL of sample is used, the nutrients must be added directly to the BOD bottle. Add 0.3 mLs of each of the following: phosphate buffer (7.1), magnesium sulfate (7.2), calcium chloride (7.3) and ferric chloride (7.4).

- Measure a proper amount of well-mixed sample into a detergent cleaned and distilled water rinsed BOD bottle. Prepare at least 3 dilutions for each sample:

  3.0 mL - 25 mL use wide bore pipette
  25 mL - 50 mL use a 50.0 mL graduated cylinder
  50 mL - 100 mL use a 100 mL graduated cylinder
  100 mL - 250 mL use a 250 mL graduated cylinder

Note: Dilutions under 3 mL must be made using serial dilutions.

11.9 Serial Dilutions

- For sample volumes between 0.30 and 3.0 make Dilution A and see examples below.

  Dilution A

  Pipette 5 mL of sample into a 50 mL graduated cylinder and dilute to 50 mL with dilution water. Pour into a disposable plastic cup labeled Dilution A.

  Sample volume needed:

0.30 mL = Pipette 3.0 mL of Dilution A into a BOD bottle.
0.50 mL = Pipette 5.0 mL of Dilution A into a BOD bottle.
1.0 mL = Pipette 10.0 mL of Dilution A into a BOD bottle.
2.0 mL = Pipette 20.0 mL of Dilution A into a BOD bottle.

- For sample volumes below 0.3 mL make Dilution B.

  Dilution B

  Pipette 5 mL of diluted sample from Dilution A to another 50 mL graduated cylinder and dilute to 50 mL with dilution water. Pour into a disposable plastic cup labeled Dilution B.

  Sample volume needed

  0.05 mL = Pipette 5.0 mL of Dilution B into a BOD bottle.
  0.10 mL = Pipette 10.0 mL of Dilution B into a BOD bottle.
  0.20 mL = Pipette 20.0 mL of Dilution B into a BOD bottle.

- Fill the rest of the bottle with dilution water, so the initial D.O. can be determined.

11.10 Initial Dissolved Oxygen Determination

- Determine the initial D.O. by inserting the probe into each bottle and read the D.O. directly from the meter when the reading has stabilized. Record the initial D.O.

Note: Rinse probe after each set of samples.

- If the initial D.O. is < 7.0 mg/L or > 9.3 mg/L shake the BOD bottle containing diluted sample until the D.O. is within range.

11.11 Incubation

- After the initial D.O. is recorded refill the BOD bottle with dilution water and insert the stopper so that all of the air is out of the bottle. Place water seal cap on bottle to reduce evaporation.

- Place samples in the incubator at 20^{o} C +/- 1^{o} C for 5 days. Make sure that each batch of bottles is labeled with the day the final D.O. is to be determined (5 days).

- To determine the final D.O. of samples insert the probe into each bottle and read the D.O. directly from the meter. Record the final D.O.

12.0 **CALCULATIONS:**

12.1 Each dilution is calculated out unless the difference between the initial and final reading is < 2.0 mg/L or the final D.O. is < 1.0 mg/L.

- o If all dilutions have a final D.O. reading of <1.0 mg/L, the following calculation is used to obtain an ESTIMATE.
- o Use smallest sample volume data.
- o (Initial D.O. - 1.0 - s.c.) x dilution factor = Est.
- o The result is reported as > Est. and qualified with an “E”.
- o All dilutions with >/= 2.0 mg/L D.O. difference are averaged for the final BOD result except when toxicity is present. If the D.O. difference between the initial and final reading is <2.0, then that BOD is not used for the average.
- o If all dilutions have D.O. differences < 2.0 mg/L, the following calculation is used to obtain an ESTIMATE.

  Use largest sample volume data.

  (2.0 - s.c.) x dilution factor = Est.

  The result is reported as < Est. and qualified with an “E”.

  Note: The above guidelines are followed as a "general" rule. These guidelines may be invalidated depending on the interpretation of each case.

  Note: As a general rule, BOD's are not to be reset since the holding time limit of 48 hours would have expired.

12.2 When seed is not used:

BOD mg/L = (initial D.O. - final D.O.) x dilution factor

Note: dilution factor = total volume (300 mL) sample volume used for BOD determination

12.3 When seed is used:

o Determine the Seed Correction of the four seed dilutions using the calculation below.

Average the four BOD results. Determine the seed correction (s.c.) by the following:

$$s.c. = \frac{A \times B}{300}$$

where:

A= the average BOD result
B= mLs of seed per 300 mL BOD bottle

- BOD mg/L = [(initial D.O. - final D.O.) - s.c.] x dilution factor

12.4 Average the BOD mg/L calculated for each dilution to get a BOD mg/L for the sample. Use this value to record the data. When recording data make sure that the proper number of significant digits is used. (See chart below)

If BOD mg/L is:

< 10 mg/L record as X.X
> 10 mg/L record as XX
> 100 mg/L record as XX0
> 1,000 mg/L record as X,X00
> 10,000 mg/L record as XX,000

12.5 Toxicity is an interpretation of the analyst and QC Technician. A sample is considered toxic if the dilutions show higher values in the more dilute concentrations. If toxicity is suspected report the result with the highest BOD (lowest sample volume).

13.0 **METHOD PERFORMANCE:**

13.1 The method performance is reviewed through the the Performance Testing Samples taken through this procedure.

13.2 See Appendix B Table I for a summary of the IDOC

14.0 **POLLUTION PREVENTION:**

14.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation. Laboratory staff should order where possible acceptable non-toxic alternative supplies and prepare only those quantities of reagents or standards that will be used prior to the expiration date. Other appropriate measures to minimize waste generation should be brought to the attention of laboratory management. All laboratory waste shall be handled as directed by the Laboratory Waste Management Plan and Hazardous Waste Contingency Plan.

15.0 **DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES:**

15.1 At a minimum there are three levels of data review:

15.2 Analyst/Technician verification at the bench top are looking at instrument performance as it relates to initial calibration, calibration verification, cleanliness.

15.3 Supervisor/Analyst verification after analysis are looking for sample concentration versus linear range of the instrument, typical patterns resulting from the compounds or elements in question, and quality control measure criteria. Data is either accepted without qualification, accepted with qualification, or rejected with samples reprocessed.

15.4 Report Reviewer/Project Management personnel look at the presentation of the data on final reports. The reports are verified for holding time compliance, receipt conditions, method citation and reasonableness, which may include field duplicate analysis, analytical comments and qualifications presented by the previous reviews, and comparing results of similar analytical techniques as it relates to the project site information when available.

15.5 A fourth level of review , data validation is determined from a project or program scope of laboratory services and encompasses the generation Quality Control summaries in the media of “Form Generation”,

15.2 See Appendix B Tables listed below for summaries of review:

Analyst/Technician Data Assessment	Table L
Analyst/Supervisor Data Assessment	Table M
Report Reviewer/Project Management Data Assessment	Table N
Corrective Actions	Table O
Contingencies for Out of Control Data	

16.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

16.1 Assessment of Quality Control measures is done to provide a level of confidence in the data generated. The measures provide documentation that the instrument conditions were reliable during the analysis.

17.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

17.1 During analysis, events occur specific to the physical and chemical characteristics of the environmental sample. When possible, with received sample volumes, data generated along with measures that do not meet statistical goals are re-analyzed again to see if the statistical goal can be achieved. When environmental samples do not meet statistical unacceptable data is generated. These events are different from those pertaining to instrument operating conditions. These events occur when the instruments are operating under ideal conditions.

17.2 See Table P for a summary.

17.3 See Table Q for a list of qualifiers.

18.0 WASTE MANAGEMENT

18.1 To minimize waste during sample preparation has two benefits. The first benefit is a cost savings to the lab in materials and supplies. The second is a benefit to the environment as less materials need to be disposed.

REFERENCES:

40CFR Part 136
American Society for Quality Control (ASQC), Definitions of Environmental Quality Assurance Terms, 1996
American National Standards Institute (ANSI), Style Manual for Preparation of Proposed American National Standards, Eighth Edition, March 1991
ANSI/ASQC E4, 1994
ANSI N42.23-1995, Measurement and Associated Instrument Quality Assurance for National Institute of Standards and Technology (NIST)
National Environmental Laboratory Accreditation Conference (NELAC), July 1998 Standards
Random House College Dictionary
US EPA Quality Assurance Division (QAD)
Webster's New World Dictionary of the American Language

FLOWCHARTS

Not Applicable

VALIDATION DATA:

The laboratory must demonstrate initial proficiency with each combination of sample preparation and determinative methods utilized, by generating data of acceptable accuracy and precision for a reference sample containing the target parameters in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made. Analysts previously trained shall perform a continued demonstration of proficiency annually by performing “Yearly Demonstration of Capability” (YDC).

The values from the determinative method are presented as guidance only and are not intended as absolute acceptance criteria. Accuracy of parameters routinely analyzed by En Chem can be found in Appendix B Initial Demonstration Data Table I. Blind studies are available upon request from the QC Manager.

MANAGEMENT APPROVAL AND REVIEW OF SOPS - POLICY AND DOCUMENTATION

REVIEWED BY: _____ 3/27/03
Section Supervisor — Date

REVIEWED BY: M. Suha 27-Mar-03
Quality Assurance Officer — Date

APPROVED BY: _____ 3/27/03
Laboratory Manager — Date

Periodic Review Record

Review Date					
Initials					

APPENDICES

Appendix A **DEFINITIONS**

Appendix B **TABLES**

Appendix A

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)
Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)
Analytical Detection Limit: the smallest amount of an analyte that can be distinguished in a sample by a given measurement procedure throughout a given (e.g., 0.95) confidence interval. (applicable only to radiochemistry)
Analyst: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)
Assessment Criteria: the measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)
Batch: a group of 20 or fewer samples that have quality control associated with them
Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)
Deficiency: an unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)
Demonstration of Capability: a procedure to establish the ability of the analyst to generate acceptable accuracy. (NELAC)
Detection Limit: the lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated degree of confidence.
Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)
Duplicate Analyses: the analyses or measurements of the variable of interest performed identically on two sub-samples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)
Equipment Blank: a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)
estimate of Target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (QAMS)
Matrix Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (QAMS)
May: denotes permitted action, but not required action. (NELAC)
Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)
Method Detection Limit: the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)
Must: denotes a requirement that must be met. (Random House College Dictionary)

National Institute of Standards and Technology (NIST): an agency of the US Department of Commerce's Technology Administration that is working with EPA, States, NELAC, and other public and commercial entities to establish a system under which private sector companies and interested States can be accredited by NIST to provide NIST-traceable proficiency testing (PT) to those laboratories testing drinking water and wastewater. (NIST)

Negative Control: measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

(items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ASQC E-41994)

Quantitation Limits: the maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

Range: the difference between the minimum and the maximum of a set of values. (EPA-QAD)

Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted. (EPA-QAD)

Reagent Blank (method blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions. (EPA-QAD)

Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Method: a method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELA*C)*

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.08)

Replicate Analyses: the measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Requirement:. denotes a mandatory specification; often designated by the term "shall". (NELAC)

Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 USC 321 *et seq.* (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)

Sample Duplicate: two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)
Selectivity: (Analytical chemistry) the capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (EPA-QAD)
Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Shall: denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (ANSI)
Should: denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI)
Spike: a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes. (NELAC)
Standard: the document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)
Standard Operating Procedures (SOPs): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)
Standardized Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)
Supervisor (however named): the individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)
Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (QAMS)
Systems Audit (also Technical Systems Audit): a thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Test: a technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method: an adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Tolerance Chart: A chart in which the plotted quality control data is assessed via a tolerance level (e.g. +/- 10% of a mean) based on the precision level judged acceptable to meet overall quality/data use requirements instead of a statistical acceptance criteria (e.g. +/- 3 sigma) (applies to radiobioassay laboratories). (ANSI)

Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 *et seq*., (1976), that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

United States Environmental Protection Agency (EPA): the federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)

Validation: the process of substantiating specified performance criteria. (EPA-QAD)

Verification: confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment. The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Work Cell: a well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented.

Appendix B

Tables

Table A
REPORTING LIMITS

COMPOUND NAME	AQUEOUS (mg/L)	
	MDL	EQL
Biochemical Oxygen Demand	--	2.0
Carbonaceous Biochemical Oxygen Demand	--	2.0

Table B

REQUIRED SAFETY EQUIPMENT

Item:	Safety Apparel Description	Mandatory for Procedure	Optional
1	Safety Goggles or Glasses	✓	
2	Lab Coat	✓	
3	Gloves	✓	
4	Face Shield		✓

Table C

EQUIPMENT

Equipment	Manufacturer	Model(s)
pH meter	sympHony	SB20
Dissolved oxygen meter	YSI	5000
Dissolved oxygen probe	YSI	5010
Incubator	Fisher	307
Incubator	VWR	2020
Incubator	Precision	815
Incubator	Precision	
Water bath	VWR	1204
Balance	OHAUS	AP1105

Table D

SUPPLIES

Supplies	Manufacturer	Vendor	Catalog #
Adjustable pipetter	Finnpipette		
Pipet helper	Brinkman	VWR	53573-000
Green Bay Met. Sewerage District domestic wastewater for seed			
50 mL graduated cylinders	Kimax	Fisher	08-548C
100 mL graduated cylinders	Kimax	Fisher	08-548D
250 mL graduated cylinders	Kimax	Fisher	08-548E
10 mL wide bore pipets	Kimax	Fisher	13-671-108D
25 mL wide bore pipets	Kimax	Fisher	13-671-108E
300 mL glass BOD pipets	Wheaton	Fisher	02-926-27
Glass microfiber filters	Fisherbrand	Fisher	09-804-90A
YSI 5906 Membrane cap kit	YSI	Fisher	13-299-77
Starch indicator paper	Precision Labs	VWR	60799-008

Table E

REAGENTS

Reagent	Purity	Manufacturer	Vendor	Catalog #
Phosphate Buffer Solution	pH 7.20 +/-0.02	Fisher	Fisher	SP341-1
Magnesium Sulfate Solution		Fisher	Fisher	SM109-1
Magnesium Sulfate Heptahydrate	100.2 %	Fisher	Fisher	M63-500
Calcium Chloride Solution		VWR	VWR	VW3308-1
Calcium Chloride Dihydrate	Certified A.C.S.	Fisher	Fisher	C79-500
Ferric Chloride Solution		VWR	VWR	VW3318-1
Ferric Chloride	Certified A.C.S.	Fisher	Fisher	I88-500
Sodium Sulfite, anhydrous	99.2%	JT Baker	JT Baker	VW3922-01
Nitrification Inhibitor (TCMP)		Hach	Hach	2533-35
Sodium Hydroxide	99.0%	JT Baker	JT Baker	VW3722-05
Sulfuric Acid	96.3%	Mallinckrodt	Mallinckrodt	2876

Table F

STANDARDS

Standard	Acronym	Concentration	Direction Found At	Alias
Phosphate Buffer Solution		pH 7.2	7.1	Nutrient
Magnesium Sulfate Solution		2.25 %	7.2	Nutrient
Calcium Chloride Solution		2.75 %	7.3	Nutrient
Ferric Chloride Solution		0.025 %	7.4	Nutrient
Sodium Hydroxide Solution		1 N	7.9	pH adjuster
Sulfuric Acid Solution		1 N	7.10	pH adjuster
Dilution Water			7.12	

Table G

SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

Matrix	Prep Method	Container(s)	Preservation	Shipment Conditions	Lab Storage Conditions
Aqueous		Clean Plastic or glass containers	Cool 4+/- 2^{o} C	On ice 4+/- 2^{o} Celsius	Cool 4+/- 2^{o} C

Table H

QUALITY CONTROL

Parameter	Frequency SM Citation
Method Blank	1 for every 10 samples
GGA Standard	At the beginning of the batch and after every 10 samples
Sample Duplicate	1 for every 10 samples by matrice

Table I

En Chem Initial Demonstration of Capability

Element	Symbol	Conc. ppb	IDC-1	IDC-2	IDC-3	IDC-4	EPA Criteria	SW846 Criteria

Not Applicable

Table J

RECOVERY LIMITS

Spike Parameter	Aqueous				Solid			
	LCS	RPD	MS	RPD	LCS	RPD	MS	RPD

Not Applicable

Table K

SURROGATE LIMITS

Spike Parameter	Aqueous	Solid
	RECOVERY %	RECOVERY %

Not Applicable

Table L

ANALYST/TACHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**Method Citation SM If these conditions are not achieved ⇒**
Initial Meter Calibration	• 1
Method Blanks	• 2
GGA Standard	• 3
Duplicate Precision	• 4
Seed Correction	• 5
Toxic Slides	• 6
Oxygen Depletion	• 7

1. Check instrumentation/equipment condition prior to use. Enter maintenance in instrument log if performed. Perform another initial calibration. No data can be reported without proper calibration.

2. In the absence of project specific requirements the D.O. difference should be no more than 0.2 mg/L. If one of the two blanks is above 0.2 mg/L the analytical results will be flagged with a “3”. Generate on Non-Conformance Memo.

3. In the absence of project specific requirements, Glucose/Glutamic Acid Standards are used to determine the quality of the dilution water and accuracy of the D.O. meter. The BOD of the standards must be within 198+/-30.5 mg/L (167.5-228.5 mg/L). . If the standard is not within the limits the analytical results affected will be flagged with a “6”. Generate on Non-Conformance Memo

4. In the absence of project specific requirements, the duplicate analysis RPD must be less than 20%. If the RPD is not within limits affected analytical results will be flagged with a “4”. Generate on Non-Conformance Memo.

5. Samples that were seeded will need the seed correction factor applied to the final calculation.

6. A sample is considered toxic if the dilutions show higher values in the more dilute concentrations. If toxicity is suspected report the result with the highest BOD

7. The difference between the initial and final D.O. reading is >2.0 mg/L. If not do not include in the calculation of the BOD. If the final D.O. reading is < 1.0 mg/L, the results will be an estimate. The analytical results will be flagged with a “7”. If the depletion was insufficient on all the dilutions, the analytical results will be flagged with a “5”.

Table M
ANALYST/SUPERVISOR DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**Method Citation SM If these conditions are not achieved ⇒**
Initial Meter Calibration	• 1
Method Blanks	• 2
GGA Standard	• 3
Duplicate Precision	• 4
Seed Correction	• 5
Toxic Slides	• 6
Oxygen Depletion	• 7

1. Verify instrumentation/equipment condition prior to use. Verify maintenance in instrument log if performed. Perform another initial calibration. No data can be reported without proper calibration.

2. In the absence of project specific requirements verify the D.O. difference should be no more than 0.2 mg/L. If one of the two blanks is above 0.2 mg/L the analytical results will be flagged with a “3”. Generate on Non-Conformance Memo.

3. In the absence of project specific requirements, Glucose/Glutamic Acid Standards are used to determine the quality of the dilution water and accuracy of the D.O. meter. Verify the BOD of the standards within 198+/-30.5 mg/L (167.5-228.5 mg/L). . If the standard is not within the limits the analytical results affected will be flagged with a “6”. Generate on Non-Conformance Memo

4. In the absence of project specific requirements, the duplicate analysis RPD must be less than 20%.Verify the RPD is within limits. Verify that affected analytical results are flagged with a “4” if not within limits. Generate on Non-Conformance Memo.

5. Verify samples that were seeded are corrected.

6. A sample is considered toxic if the dilutions show higher values in the more dilute concentrations. If toxicity is suspected report the result with the highest BOD

7. Verify the difference between the initial and final D.O. reading is >2.0 mg/L. If not do not include in the calculation of the BOD. If the final D.O. reading is < 1.0 mg/L, the results will be an estimate. The analytical results will be flagged with a “7”. If the depletion was insufficient on all the dilutions, the analytical results will be flagged with a “5”.

Table N
REPORT REVIEWER/PROJECT MANAGEMENT DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**Method Citation SM If these conditions are not achieved ⇒**
Holding Time Compliance	• 1
Sample Receipt Conditions and Preservation	• 2
Method Citation	• 3
Reasonableness: Duplicate	• 4
Reasonableness: Analytical Comments Qualifications	• 5

1. Flag results with an H and the number of days past hold in parenthesis e.g. H(5).

2. In prose describe the receipt conditions as they relate to the acceptance criteria list in Table G

3. Compare to regulatory program in QAPjP or Chain of Custody.

4. Are duplicate with 20% agreement. If not, internally flag for review.

5. Do related suites of tests agree or are reasonable.

Table O
CORRECTIVE ACTIONS

Analytical Method Acceptance Criteria⇨ Quality Control Measure ⇩	**Method Citation SM If these conditions are not achieved ⇒**
Method Blank	< 0.2 mg/L
GGA Standards	Within Control Limits

1. See section 10 for procedures to correct.

2. See section 10 for procedures to correct..

Table P
CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Analytical Method Acceptance Criteria⇨ Quality Control Measure ⇩	**Method Citation SM If these conditions are not achieved ⇒**	**Corrective Action (Key below)**
Method Blank	• < 0.2 mg/L	• 1
GGA Standards	• Within Limits	• 1
Duplicates	• Within Limits	• 1

1. Flag appropriately all samples affected.

Table Q
DATA QUALIFIERS

B	The analyte has been detected between the method detection limit and the reporting limit.
C	Elevated detection limit due to matrix effects.
H(n)	Preservation or analysis performed “n” days past holding time (See Sample Narrative).
K	Sample received unpreserved. Sample was either preserved at the time of receipt or at the time of sample preparation.
L	Elevated detection limit due to low sample volume.
&	Laboratory Control Spike recovery not within control limits.
*	Duplicate analyses not within control limits.
1	Dissolved analyte or filtered analyte greater than total analyte; analyses passed QC based on precision criteria.
2	Dissolved analyte or filtered analyte greater than total analyte; analyses failed QC based on precision criteria. (See Sample Narrative).
3	BOD result is estimated due to the BOD blank exceeding the allowable oxygen depletion.
4	BOD duplicate precision not within control limits. Due to the 48 hour holding time for this test, it is not practical to reanalyze and try to correct the deficiency.
5	BOD result is estimated due to insufficient oxygen depletion. Due to the 48 hour holding time for this test, it is not practical to reanalyze and try to correct the deficiency.
6	BOD laboratory control sample not within control limits. Due to the 48 hour holding time for this test, it is not practical to reanalyze and try to correct the deficiency.
7	BOD result is estimated due to complete oxygen depletion. Due to the 48 hour holding time for this test, it is not practical to reanalyze and try to correct the deficiency.